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LA UR-84-541

CONF-840944--1

LA-UR--84-541

DE84 007486

TITLE: Ceramic-Ceramic Seals by Microwave Heating

AUTHOR(S): T. T. Meek and R. D. Blake

SUBMITTED TO: The 14th European Microwave Conference, Liege, Belgium
For presentation, and publication in conference proceedings.

Los Alamos Los Alamos National Laboratory
Los Alamos, New Mexico 87545

Ceramic-Ceramic Seals by Microwave Heating .

by

T. T. Meek and R. D. Blake
Materials Science and Technology Division
Los Alamos National Laboratory
Los Alamos, NM 87545

I. Introduction

Thermal processing by microwave heating is not a new idea, but one that has been around for two decades. Early work by Von Hippel[1,2] on the interaction of ceramic and glass materials with UHF electromagnetic radiation initiated interest in this form of ceramic thermal processing.

Tinga[3-8] began investigating UHF heating of ceramics and glasses at the University of Alberta, Canada in the mid 1960's and has been active in this area up to the present. General Motors Corporation in the late 1960's also became involved in this area.

This paper addresses work done on fabrication of ceramic-glass-ceramic seals using microwave heating. Seals were made using Minnesota Mining and Mfg. (3M) alumina substrates of 96w% Al_2O_3, 3w% SiO_2 and 1w% MgO. Sealing glasses used were Owens Illinois Glass codes OI-1755C, OI-0038, and OI-1613.

II. Experimental

All thermal processing by microwaves was done using a Lit on model 1520 microwave oven. This oven operates at 2.45 GHz with a power output of 700 watts. Table I lists the various ceramic-glass-ceramic systems fabricated and table II lists the microstructural analysis techniques used. Figure I shows the reaction cavity and the area in the microwave oven used for thermal processing. The cavity measured 0.32 cm by 7.62 cm x 7.62 cm. The cavity was insulated with 5.08 cm of Union Carbide Corp. Zircar 15 insulation.

Table I

Glass	Fusion Temp.	Ceramic	Processing Time	Processing Technique	Power Used
OI-1756C	462°C	3M	100.0 min.	Microwave	0.85 kwhr
OI-1756C	462°C	3M	15 hrs	Conventional	15.00 kwhr
OI-0038	735°C	3M	100.0 min.	Microwave	0.85 kwhr
OI-0038	735°C	3M	15 min.	Microwave	.13 kwhr
OI-0038	735°C	3M	5 min.	Microwave	.04 kwhr
OI-0038	735°C	3M	30 min.	Microwave	.26 kwhr
OI-1613	1450°C	3M	100.0 min.	Microwave	.85 kwhr
OI-1613	1450°C	3M	30 min.	Microwave	.26 kwhr
OI-1613	1450°C	3M	15 min.	Microwave	.13 kwhr
OI-1613	1450°C	3M	15 min.	Microwave	.13 kwhr

Table II

Microstructural Analysis

Analysis Technique	Data Obtained
SEM	Interface characteristics
Photomicrograph	Interface characteristics
EDAX	Diffusion of reactants
	Composition of seal

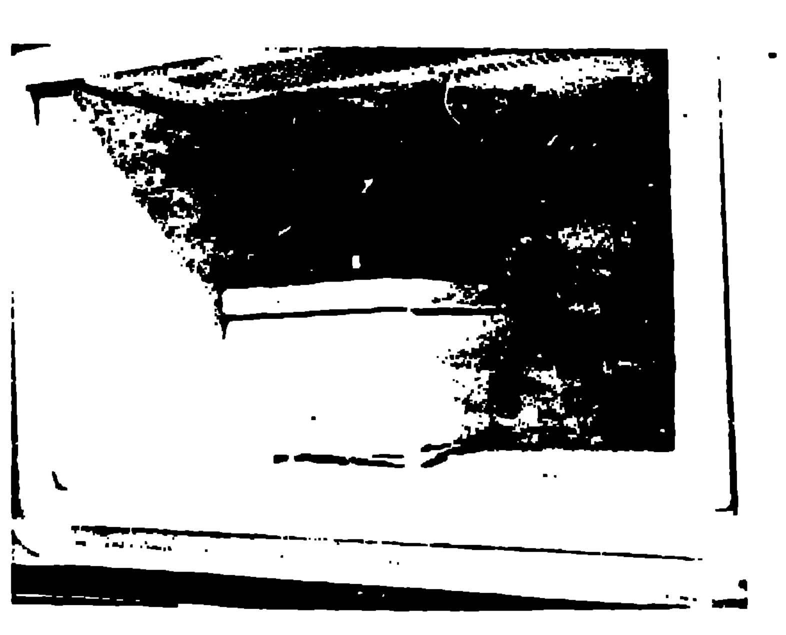


Figure I - Microwave area with reaction cavity inside.

Samples to be thermally processed were fabricated using 0.051 cm x 5.08 cm x 5.08 cm. 3M 96% alumina substrates with one of the glasses listed in Table I between them.

A typical sample is shown in figure 2.

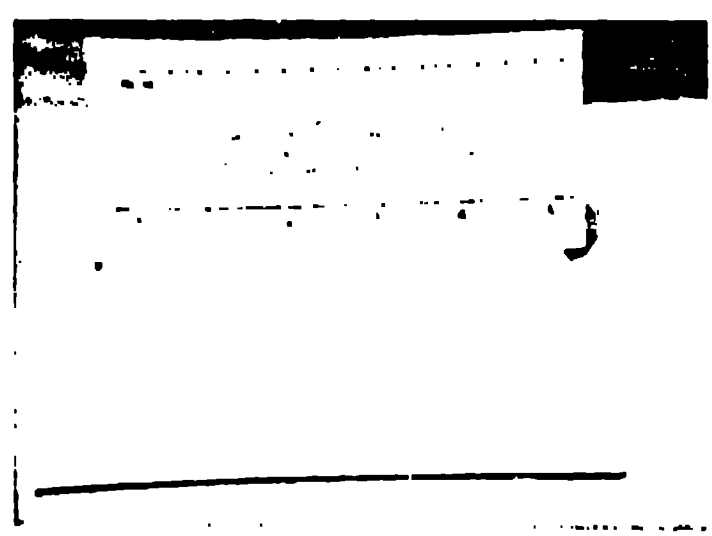


Figure II - Shown is a typical geometry heated using UHF Microwave Energy. The sample is made of Al_2O_3 substrates with glass in between.

After the glass slurry was made, it was spread over the top surface of the bottom substrate. The top substrate was then placed on the slurry and the combination was placed into the reaction cavity. The microwave oven was then turned on and the timer set to the desired processing time. Processing temperature data were obtained using a Leeds and Northrup optical pyrometer focused on the front edge of the sample, and figure III shows typical time temperature profiles obtained for 0I1613 glass. Heating rates varied from 1000°C/hr to over 30,000°C/hr. After the run was completed the sample was then examined using metallographic, SEM, and EDAX techniques

III. Discussion

It is at present unclear in what manner electromagnetic radiation couples with the sealing materials listed in table I. What is clear however, is that in order for the compositions listed in Table I to enter into a reaction resulting in the microstructures of Figures IV-VIII, certain processes had to occur. As is known, most oxide materials will not couple to 2.45 GHz radiation so; therefore, a coupling agent was added to the glass slurry to allow initial coupling. The coupling agent may be organic or inorganic; however, it must contain C = C, NO_3, H_2O or other bonds that will couple to the electromagnetic radiation and heat the ceramic-glass combination to a temperature of a few hundred degrees C (~300°C). At this temperature the glass slurry will begin to exhibit high frequency relaxation mechanisms such as ionic conduction and molecular vibration which will enable the glass to couple to 2.45 GHz radiation. At this point the

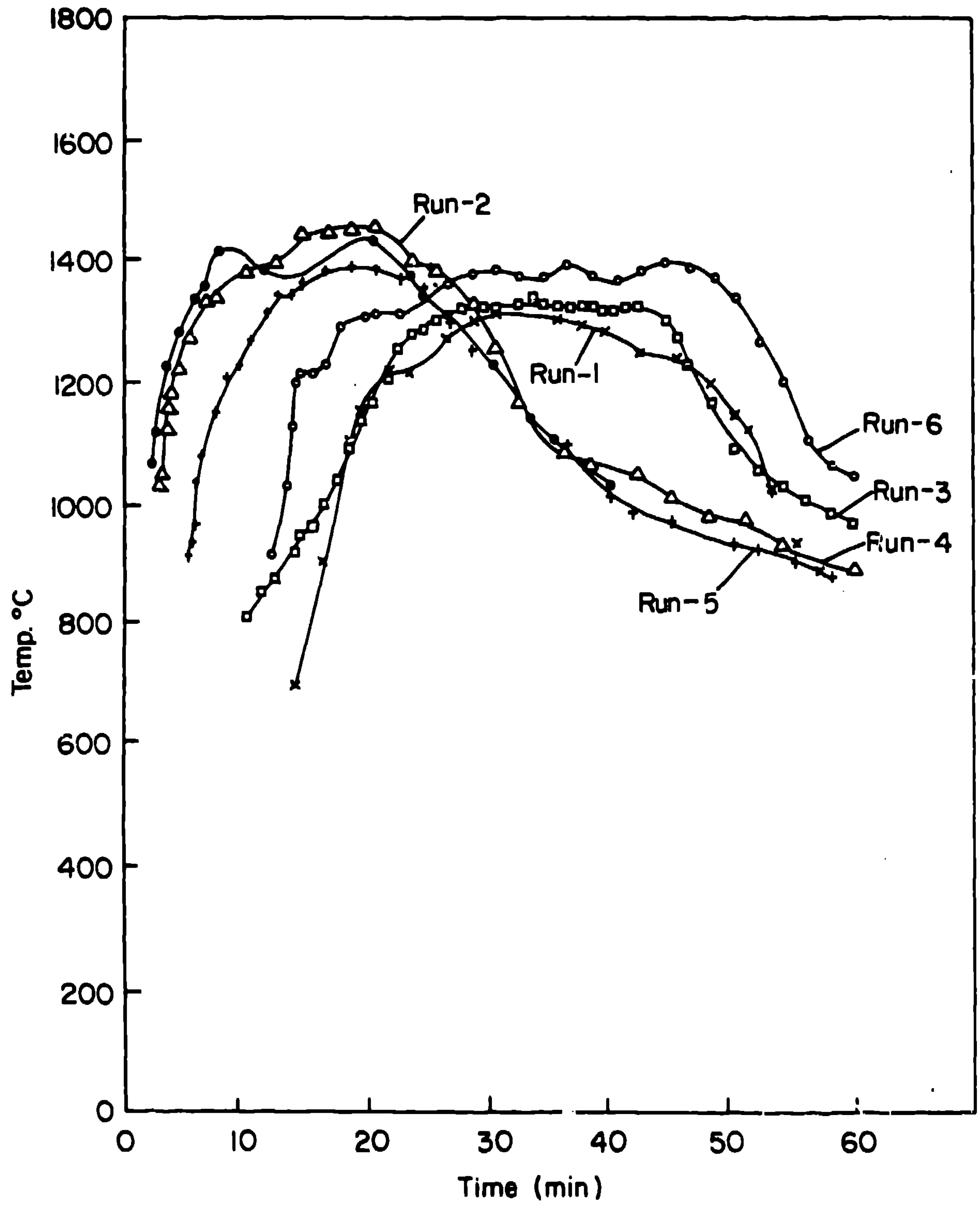


Figure III Time-temperature curves for OI-1613 sealing glass used for fabrication of ceramic-ceramic seal by UHF microwave heating temperature of the system will increase. As evidenced by the time-temperature curves of figure III, the reaction will proceed, and upon completion, temperature will begin to decrease due to decoupling of the new phase to the microwave field. Under the influence of the electromagnetic field present in the microwave reaction cavity, reaction kinetics may differ from kinetics in normal thermal convection heating. This may explain differences seen in the respective microstructures.

IX. Results

Figures IV through VIII show photomicrographs and EDAX scans of various seals made by microwave heating and conventional heating. Note the difference in glass-ceramic interface characteristics. Diffusion bonding is evident and is the predominant bonding mechanism with all of the seals made by microwave heating. For the seals made by conventional heating, glass-ceramic wetting is the predominant bonding mechanism. EDAX scans in figure VIII clearly show the extent of interdiffusion taking place with the microwave heating technique. The starting OI-1756 seal glass composition is 64.9w% PbO, 14.4w% ZnO, 2.4w% SiO_2 and 9.4w%, B_2O_3. This is the sealing glass used in figures IV and V and analyzed in Figure VIII. Very little interdiffusion is seen in the conventionally heated seals while extensive interdiffusion is seen in the seals fabricated using microwave heating.

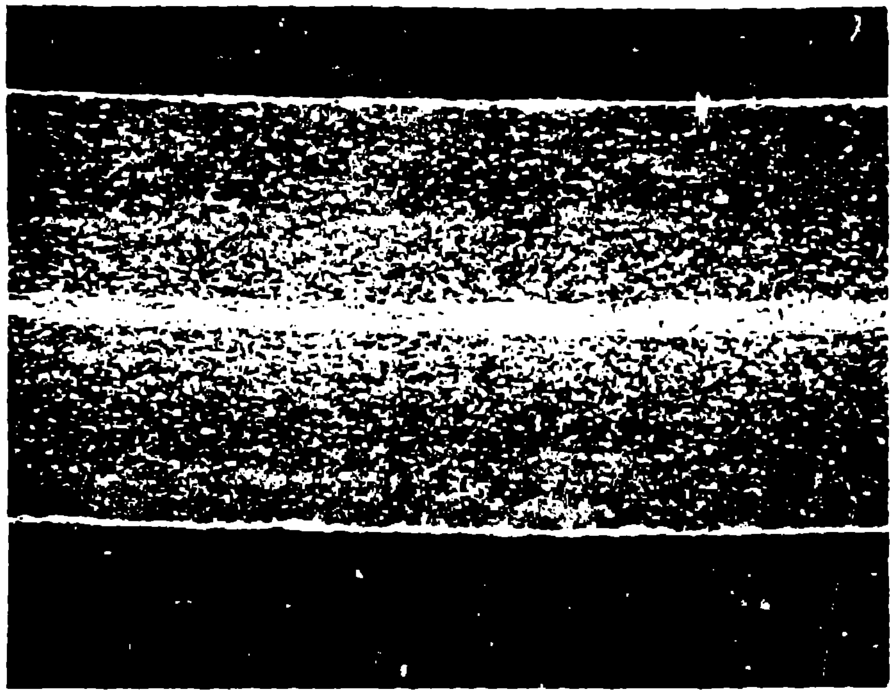
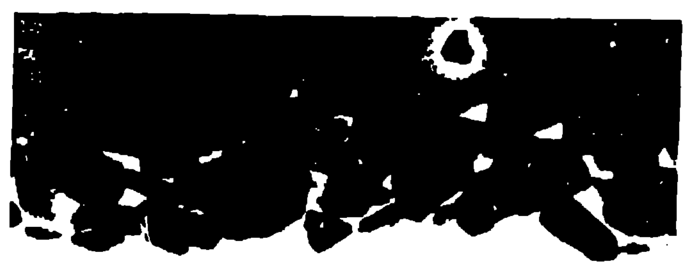


Figure IV - Photomicrograph (45X) of 462°C seal by microwave heating. Regions I and III are the substrates and region II is the sealing glass OI-1756.

The EDAX scans in figure VIII identify the composition of the glassy material (in figure IV) between the two alumina substrates heated using UHF microwave energy. The larger grayish glassy phase in figure IV is shown to be composed of lead oxide, alumina, and a small amount of silica. Alumina is the primary material in this region. The smaller darker glassy phase is composed of alumina and zinc. Again alumina is the predominate material in this region. While the original glass contained no alumina the resultant glassy matrix contained mostly alumina. This serves to point out the extent of interdiffusion resulting from microwave heating.

Figure IX shows the energy used in fabricating the 462°C glass-ceramic seal by microwave heating and by conventional heating. Clearly fabricating the seal by microwave heating is more economical both in energy savings and in labor costs.



I

II

Figure V - Photomicrograph (2000X) of 462°C seal made by conventional heating. Region I is the 3M alumina substrate and Region II is the OI-1756 sealing glass.

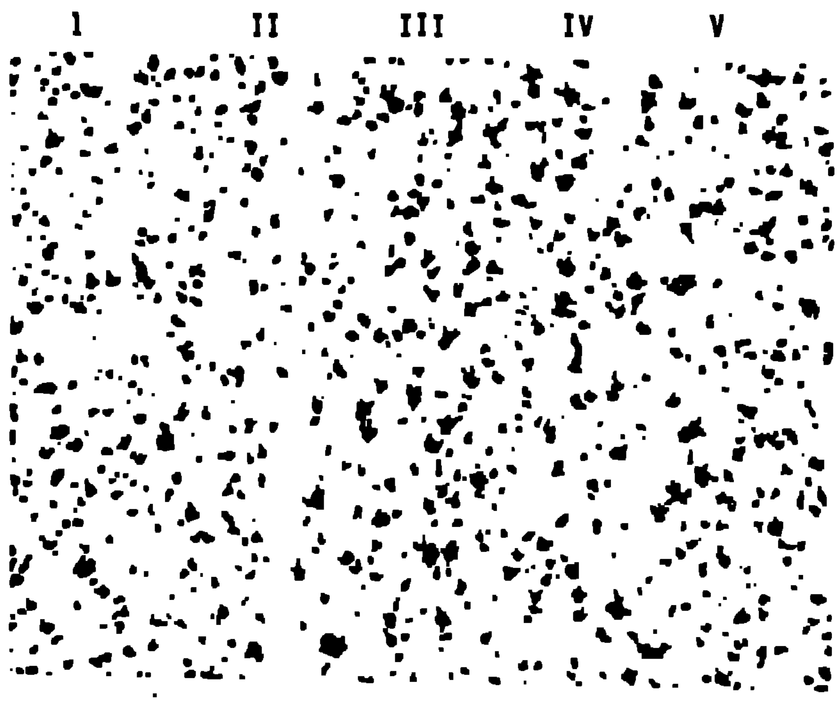


Figure VI - Photomicrograph (75X) of 735°C seal made by microwave heating. Regions I, III, and V are the alumina substrates and regions II and IV are the glass seals made from OI-0038 glass.

Figure VII - Photomicrograph (250X) of 1450°C seal made by microwave heating. Regions I and III are the alumina substrates and region II is the glass seal made from OI-1613 glass.

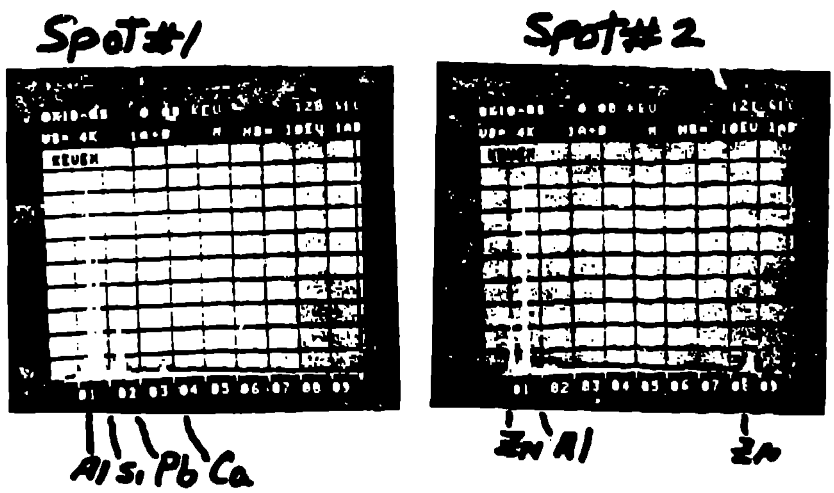


Figure VIII - EDAX scans of 462°C seal made by microwave heating.

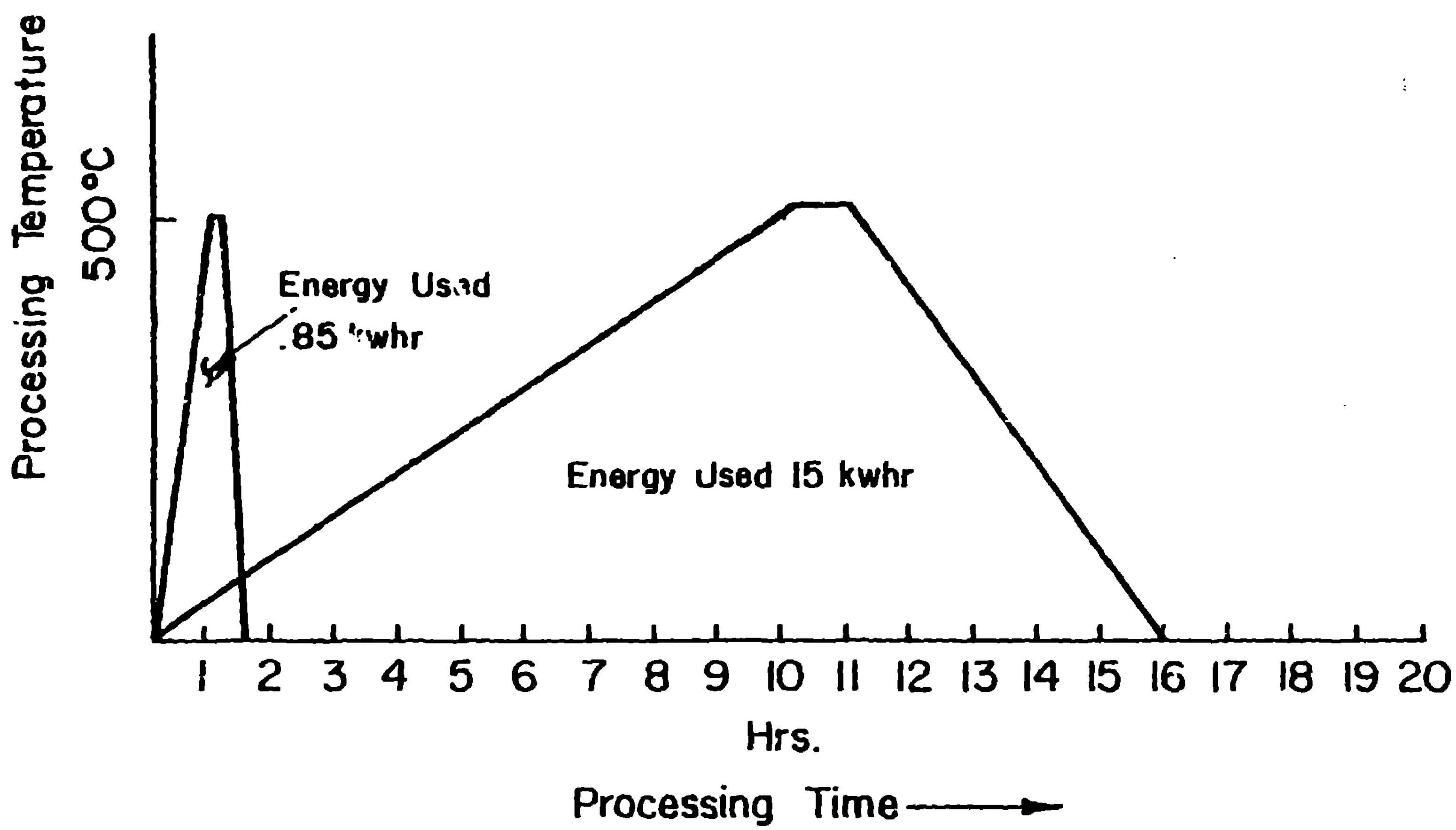


Figure IX - Energy usage comparison between microwave formed seal of figure IV and conventionally formed seal of figure V.

V. Conclusion

Several conclusions may be drawn from this work. First, less energy is required to form a glass-ceramic seal by microwave heating than by conventional heating. Second, less time is required to form the seal by microwave heating, and third, the seal composition is different, and the bonding is different. The microstructure of the seal formed by microwave heating reflects extensive diffusion of the glass constitients throughout the alumina substrate; and alumina throughout the seal glassy matrix. Fourth, higher heating rates are possible with microwave heating than with conventional heating. Last, the energy is coupled differently to the reactants using microwave heating, and thus the reaction kinetics may be different, as indicated by the vastly different microstructures obtained.

VI. Acknowledgements

The authors would like to thank Mr. H. Sheinberg for his help in the preparation of this paper and for his helpful discussions on the content of this paper.

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